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# **KINGDOM OF THE NETHERLANDS**

## **INDUSTRIAL PROPERTY OFFICE**

It is certified herewith that in The Netherlands on December 5, 2002 under number 1022087,  
in the name of:

DRAKA FIBRE TECHNOLOGY B.V.

at Eindhoven

a patent application was filed in respect of:

"Method of manufacturing an optical fibre.",

and that the documents attached hereto entirely agree with the documents originally filed in  
support of said application.

Rijswijk, December 8, 2003.

The Director of the Industrial Property Office,  
in his place,

Ms. I.W. Scheevelenbos-de Reus

## Method of manufacturing an optical fibre.

5 The present invention relates to a method of manufacturing an optical fibre by carrying out one or more a chemical vapour deposition reactions in a substrate tube, which method comprises the following steps:

i) supplying one or more doped or undoped glass-forming precursors to the substrate tube,

10 ii) supplying a stoichiometric excess of oxygen to the substrate tube,

15 iii) setting up a reaction in the substrate tube between the reactants supplied in steps i) and ii) so as to effect the deposition of one or more glass layers on the interior of the substrate tube,

iv) subjecting the substrate tube thus coated in step 20 iii) to a collapsing process so as to form a preform, and finally

25 v) drawing said preform into an optical fibre while heating the preform and subsequently cooling said optical fibre.

Such a method is known per se from US patent No. 4,314,833, in which the deposition rate is increased without fine glass particles 30 being formed in the gas phase, and in which the inside diameter of the substrate tube is kept as small as possible after the deposition process in order to guarantee a trouble-free collapse. According to said US

35 patent, it has surprisingly been found that a simple relationship exists between the inside diameter of the substrate tube during the deposition process and the maximum deposition rate at which no fine glass particles are formed in the gas phase. Three separate regions are distinguished in connection with the deposition process, viz. a first region, in which fine glass particles are formed in the gas phase, a second region, in which the lower limit for the inside diameter of the substrate tube at a 40 specific deposition rate is indicated, and finally a third region between said first and said second region, which third region defines the pre-

ferred process conditions. It can furthermore be noted in this connection that the substrate tube to be coated with glass layers and the thickness of the layers to be coated by means of a deposition process must be selected such that the subsequent collapse treatment will not cause any  
5 additional problems. The fact is that if the inside diameter of the substrate tube at the end of the deposition process is too large, certain problems may occur during the collapse treatment, an elliptical preform may be formed, for example, or a preform that sags or exhibits other geometric faults, in connection with which it is indicated that the  
10 shorter the duration of the collapse treatment, the greater the chance that a certain degree of rotational symmetry in the preform will be obtained.

One drawback of the aforesaid US patent specification as pointed out by the present inventors is the fact that it only shows  
15 experimental results of the position rates varying from 0.08 g/min to 0.45 g/min, which values are insufficiently high these days. In addition to that, the diagram that is shown in the aforesaid US patent specification does not provide any information with regard to higher deposition rates, for example deposition rates of more than 2.0 g/min. Consequently,  
20 the present inventors have found that the process conditions suggested in the aforesaid US patent specification no longer apply at deposition rates of more than 2 g/min.

One object of the present invention is thus to provide a method of manufacturing an optical fibre, which method precisely defines  
25 the process conditions required for high deposition rates.

Yet another object of the present invention is to provide a method of manufacturing an optical fibre, which method defines process conditions such that the formation of fine glass particles in the gas phase is prevented through the use of a high deposition rate.

30 Yet another object of the present invention is to provide a method of manufacturing an optical fibre, which method leads to a sub-

strate tube geometry following the deposition of one or more glass layers onto the interior of the substrate tube such that the collapsing step that follows will result in a rotationally symmetrical preform.

5 The method as referred to in the introduction is characterized in that the Reynolds number is in accordance with the formula  $64 < Re < 180$  during the deposition process according to step iii), wherein the Reynolds number is calculated on the basis of the reactants supplied to the substrate tube in step i) and step ii), under the temperature and pressure conditions that prevail in the interior of the substrate tube during step iii).

10

15 The objectives of the present invention are accomplished by carrying out the method as described above. The Reynolds number (Re) is a dimensionless parameter known to those skilled in this field of the art, which is composed of the product of the density, the gas flow rate and the inside diameter of the substrate tube divided by the viscosity, wherein the values are based on the gases supplied to the interior of the substrate tube, in particular the physical values associated with the mixtures of gases. The significance of the value of the Reynolds number in the present method will follow from the examples and comparative 20 examples that are given herein.

In the present method it is in particular preferable to use a pressure of 4-35 mbar during step iii).

In addition to that it is preferable for the substrate tube to have a temperature of 1000-1150 °C during step iii).

25 It is also desirable to use a stoichiometric excess of oxygen of 1.8-5.0 during step ii).

30 Preferably, a plasma is generated in the interior of the substrate tube for initiating a reaction between the reactants supplied in steps i) and ii) in order to thus effect the deposition of one or more glass layers thereon, wherein it is preferred to move the plasma zone with respect to the substrate tube during step iii). Thus it is prefera-

ble to carry out the present method according to the Plasma Chemical Vapour Deposition (PCVD) process.

Surprisingly, the present inventors have discovered that it is not the deposition rate that is in particular suitable for characterising the desired process conditions, as suggested in US patent specification No. 4,314,833 as discussed above, which parameter is in fact a derived quantity, but rather the total gas flow rate, viz. the glass-forming gases supplied to the interior of the substrate tube.

If a substrate tube having a temperature of less than 1000 °C is used in during step iii), undesirable incorporation of chlorine in the deposited glass layers will take place. On the other hand, if a substrate tube temperature of more than 1150 °C is used, this will lead to deformation, in particular so-called unroundness, of the substrate tube. With regard to the pressure within the substrate tube during step iii) of the present invention it applies that many impurities are incorporated in the deposited glass layers when a value higher than 35 mbar is used, which is undesirable. In particular the incorporation of chlorine will lead to problems, especially because bubbles are formed. On the other hand, if the pressure within the substrate tube is lower than 4 mbar during step iii) of the present invention, the plasma will become longer and the plasma intensity will decrease at a constant high-frequency capacity, which will result in an insufficient conversion of chlorides into oxides. As regards the stoichiometric excess of oxygen used in the present method it applies that an excess of oxygen of less than 1.8 will lead to problems with regard to the stability of the plasma, resulting in undesirable incorporation of chlorine in the deposited layers. On the other hand, if a stoichiometric excess of oxygen of more than 5.0 is used, the gas flow rate in the plasma will be too high, so that the residence time of the reactive gases in the reaction zone will be too short and the conversion efficiency will thus decrease undesirably. Moreover, undesirable formation of fine class particles will

occur. In this connection it may furthermore be noted that the coupled plasma capacity required for the deposition process is directly related to the amount of glass-forming molecules being supplied to the interior of the substrate tube.

5 The present inventors have thus found that in order to obtain an  $\text{SiO}_2$  (and any dopants) incorporation efficiency of more than 90%, the Reynolds number must not exceed 180 so as to prevent the formation of fine class particles, in particular in the case of a maximum stoichiometric excess of oxygen. The present inventors have furthermore  
10 found that the Reynolds number must be at least 64, because the plasma will exhibit a considerable degree of instability if said value is lower, in particular in the case of a minimum stoichiometric excess of oxygen.

15 The appended Figure shows the so-called working range or process window for PCVD, wherein the inside diameter of the substrate tube (mm) is plotted on the horizontal axis and the total flow rate of the gases being supplied to the interior of the substrate tube is plotted on the vertical axis. In order to obtain a deposition efficiency of more than 90%, at a deposition rate of more than 2 g/min, the working range, within which the aforesaid requirements are met, preferably lies between  
20 the upper line, which corresponds to a Reynolds number of 180, and the lower line, which corresponds to a Reynolds number of 64. The appended Figure also shows a minimum deposition rate of 2 g/minute.

25 The present invention will be explained in more detail hereinafter by means of a number of examples and comparative examples, in which connection it should be noted, however, that the present invention is by no means limited to such special examples.

Example 1.

30 A glass substrate tube having an inside diameter of 26 mm was internally coated with layers of  $\text{SiO}_2$  and  $\text{GeO}_2$ -doped  $\text{SiO}_2$ . Said layers were deposited by means of a PCVD technique, using an overall deposition rate of 2.5 g/min. The excess of oxygen amounted to 3.5, and the maximum

total gas flow through the interior of the substrate tube was set at 4196 sccm. The minimum pressure in the substrate tube during the deposition of the layers was maintained at a value of more than 10 mbar, and the maximum pressure in the substrate tube during the deposition process was 5 maintained at a value of less than 30 mbar. At the start of the deposition process, at which point the inside diameter of the substrate tube was 26 mm, the Reynolds number for the gas mixture was 113 at a temperature of 1100 °C inside the substrate tube. The deposition of the layers was stopped once the inside diameter of the substrate tube amounted to 18 10 mm, at which point the Reynolds number was 163. Following that, the tube was contracted into a massive preform, from which a high-quality optical fibre was obtained.

Examples 2-3 and comparative examples 1-2.

Substrate tubes having an inside diameter of 20 mm were 15 internally coated with  $\text{SiO}_2$  for 1 hour, using a PCVD technique. The amount of  $\text{SiCl}_4$  that was added amounted to 933 sccm each time (sufficient for a deposition rate of at least 2.5 g/min with a deposition efficiency of 100%). The excess of oxygen was varied with each new experiment, so that also the total flow and the Reynolds number varied with each new 20 experiment. The other conditions (the pressure and the temperature) were maintained at the same values as in the above Example 1. At the end of each experiment the deposition efficiency was calculated on the basis of the measured weight increase of the substrate tube. The results are shown in the Table below.

Example	O <sub>2</sub> excess	Reynolds number	Deposition efficiency
Example 2	3	136	95%
Example 3	4.5	169	93%
Comparative example 1	5	181	89%
Comparative example 2	6	209	80%

5 From the Table that is shown above it follows that the deposition efficiency is lower than 90 percent if the Reynolds number is  
10 higher than 180, which value is critical, therefore.

Comparative examples 3-4.

15 A substrate tube having an inside diameter of 29 mm was coated with glass layers, using a PCVD technique and a constant flow of SiCl<sub>4</sub> of 784 sccm. In Comparative Example 3, the excess of oxygen was slowly decreased from 5 to 1.5. Instabilities occurred in the plasma when  
20 the excess of oxygen amounted to 1.8, at which point the Reynolds number was 64, which instabilities manifested themselves in the form of serious pressure variations in the substrate tube. A further decrease of the excess of oxygen finally led to the plasma being extinguished.

20 In Comparative Example 4, the above experiment was repeated, in this case using a substrate tube having an inside diameter of 37 mm and a constant flow of SiCl<sub>4</sub> of 933 sccm. In this Comparative Example, too, the plasma was unstable when the excess of oxygen amounted to 2 or less, at which point the Reynolds number was 64.

## CLAIMS

1. A method of manufacturing an optical fibre by carrying out one or more a chemical vapour deposition reactions in a substrate tube, which method comprises the following steps:

5                   i) supplying one or more doped or undoped glass-forming precursors to the substrate tube,

10                  ii) supplying a stoichiometric excess of oxygen to the substrate tube,

15                  iii) setting up a reaction in the substrate tube between the reactants supplied in steps i) and ii) so as to effect the deposition of one or more glass layers on the interior of the substrate tube,

20                  iv) subjecting the substrate tube thus coated in step iii) to a collapsing process so as to form a preform, and finally

25                  v) drawing said preform into an optical fibre while heating the preform and subsequently cooling said optical fibre, characterized in that the Reynolds number is in accordance with the formula  $64 < Re < 180$  during the deposition process according to step iii), wherein the Reynolds number is calculated on the basis of the reactants supplied to the substrate tube in step i) and step ii), under the temperature and pressure conditions that prevail in the interior of the substrate tube during step iii).

30                  2. A method according to claim 1, characterized in that a pressure of 4-35 mbar is used during step iii).

25                  3. A method according to any one or more of the preceding claims, characterized in that the substrate tube has a temperature of 1000-1150 °C during step iii).

30                  4. A method according to any one or more of the preceding claims, characterized in that a stoichiometric excess of oxygen of ranging from 1.8-5.0 is used during step ii).

5. A method according to any one or more of the preceding

claims, characterized in that step iii) comprises the forming of a plasma within the substrate tube so as to effect the deposition of one or more glass layers.

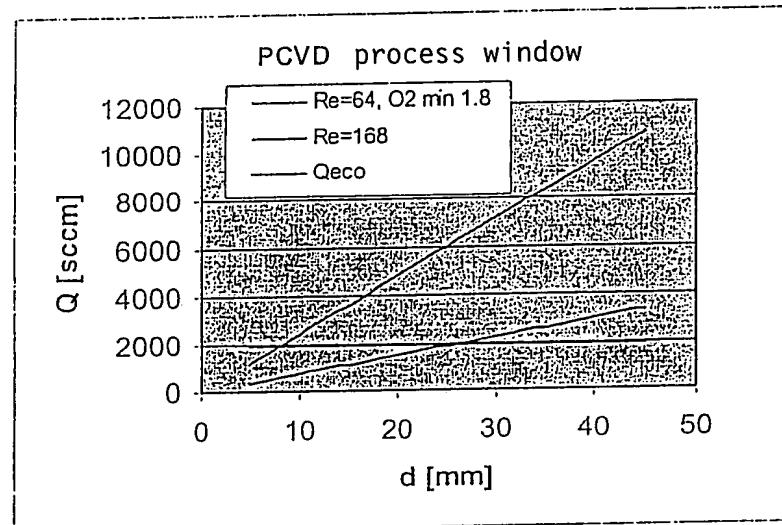
6. A method according claim 5, characterized in that the plasma zone is moved with respect to the substrate tube during step iii).

5 7. A method according to any one or more of the preceding claims, characterized in that a deposition rate of at least 2 g/min is used in step iii).

## A B S T R A C T

The present invention relates to a method of manufacturing an optical fibre by carrying out one or more a chemical vapour deposition reactions in a substrate tube, which method comprises the following steps:

- i) supplying one or more doped or undoped glass-forming precursors to the substrate tube,
- ii) supplying a stoichiometric excess of oxygen to the substrate tube,
- iii) setting up a reaction in the substrate tube between the reactants supplied in steps i) and ii) so as to effect the deposition of one or more glass layers on the interior of the substrate tube,
- iv) subjecting the substrate tube thus coated in step iii) to a collapsing process so as to form a preform, and finally
- v) drawing said preform into an optical fibre while heating the preform and subsequently cooling said optical fibre.



Figure